

Thermo-Catalytic Cracking of Used Engine Oil into Gasoline-Range Hydrocarbons Over Na/K/Ca-Promoted Fe_3O_4 Nanoparticles Supported on HZSM-5

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Abstract

Original Research Article

The sustainable management of used engine oil (UEO) represents a critical environmental and energy challenge due to its complex composition, high toxicity, and improper disposal practices. In this study, thermo-catalytic cracking was employed as an effective route for upgrading UEO into gasoline-range hydrocarbons using Na/K/Ca-promoted Fe_3O_4 nanoparticle on HZSM-5 support as catalysts. Na- Fe_3O_4 /HZSM-5, K- Fe_3O_4 /HZSM-5, and Ca- Fe_3O_4 /HZSM-5 catalysts were evaluated over a reaction temperature range of 350–450 °C, varying catalyst loadings and reaction times to elucidate the effects of process parameters on product distribution and fuel quality. Process optimization was performed using the Taguchi experimental design coupled with analysis of variance (ANOVA), which revealed reaction temperature as the dominant parameter influencing UEO conversion, gasoline-range (C_4 – C_{12}) selectivity, and PONA composition. The highest gasoline-range selectivity of 97.91% was achieved at 450 °C using 5 wt.% Na- Fe_3O_4 /HZSM-5, attributed to the optimal balance of Brønsted and Lewis acid sites and improved accessibility of the HZSM-5 pore structure. Higher loadings resulted in reduced selectivity due to nanoparticle aggregation and partial pore blockage. The upgraded liquid products exhibited significant improvements in fuel properties compared to raw UEO, including reduced kinematic viscosity and carbon residue, specific gravity, high research octane numbers, and acceptable flash point and autoignition characteristics. These results confirm the production of gasoline-like fuels with enhanced combustion performance and handling safety. Overall, this work demonstrates the technical viability of Na/K/Ca-promoted Fe_3O_4 /HZSM-5 catalysts for efficient conversion of UEO into high-value transportation fuels, offering a promising waste to energy pathway that addresses both environmental remediation and sustainable fuel production.

Keywords: Used engine oil, Catalyst promoter, Cracking, Gasoline, Hydrocarbons.

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1.0 INTRODUCTION

The rapid growth of transportation and industrial activities has led to a continuous increase in the consumption of lubricating oils, consequently generating large volumes of used engine oil (UEO) as hazardous waste (Sánchez-Alvarracín *et al.*, 2021). UEO contains a complex mixture of degraded hydrocarbons, additives, polycyclic aromatic compounds, and other toxic constituents that pose significant environmental and public health risks when improperly disposed (Mishra *et al.*, 2021; Muhibat *et al.*, 2021; Gong *et al.*, 2023). Conventional disposal practices, such as landfilling, uncontrolled burning, or direct discharge into soil and water bodies, contribute to severe ecological degradation and long-term contamination (Mishra *et al.*, 2021).

Therefore, the development of sustainable and efficient technologies for the valorisation of UEO into useful energy products has become an urgent global priority.

Thermo-catalytic cracking of UEO has emerged as a promising pathway for converting waste hydrocarbon streams into value added fuels under relatively moderate operating conditions (Alavi *et al.*, 2019; Mishra *et al.*, 2021; Balboul *et al.*, 2022; Moses *et al.*, 2023). Unlike simple thermal processing, the presence of a suitable catalyst enhances reaction selectivity, lowers activation energy, and improves product quality by promoting controlled bond scission and secondary reactions (Santhoskumar and Ramanathan, 2020; Mishra *et al.*, 2021; Balboul *et al.*,

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2022). In particular, catalytic processing enables the selective conversion of high molecular weight hydrocarbons in UEO into lighter fractions within the gasoline boiling range, while minimizing coke formation and undesirable heavy residues (Mishra *et al.*, 2021; Osei *et al.*, 2021). This approach aligns with circular economy principles by transforming hazardous waste into clean energy carriers.

Among the various catalysts investigated for hydrocarbon conversion to fuel, HZSM-5 zeolite has attracted considerable attention due to its unique physicochemical properties (Wei *et al.*, 2017; Zahara *et al.*, 2018). HZSM-5 possesses a medium-pore, shape selective framework, high surface area, strong Brønsted acidity, and excellent thermal stability, all of which are crucial for effective cracking, isomerization, oligomerization, and aromatization reactions (Zhang *et al.*, 2020; Altalhi *et al.*, 2022; Mousavi *et al.*, 2022). Its well-defined pore structure favours the formation of gasoline-range hydrocarbons (C_5 – C_{12}) with high octane numbers, while suppressing excessive formation of heavy by-products (Vu and Armbruster, 2019; Valle *et al.*, 2022; Singh *et al.*, 2023). Moreover, HZSM-5 has demonstrated remarkable resistance to deactivation compared to amorphous catalysts, making it suitable for processing complex feedstocks such as UEO (Zhang *et al.*, 2024).

Iron oxides on the other hand are among the most technologically significant transition metal oxides owing to their extensive utilization across scientific and industrial applications (Shah *et al.*, 2025). Iron-based catalysts have attracted sustained research interest due to their versatility in catalysis, environmental compatibility, and favourable economic attributes (Shah *et al.*, 2025). Compared with many alternative catalytic materials, iron-based catalysts exhibit relatively low toxicity, potential biodegradability, and abundant natural availability, rendering them attractive for large-scale and sustainable applications (Campos *et al.*, 2015; Zhou *et al.*, 2025). At the nanoscale, iron-based materials display physicochemical properties that differ markedly from those of their bulk counterparts, which significantly enhances their catalytic performance (Shah *et al.*, 2025; Zhou *et al.*, 2025). Iron-based nanomaterials typically possess high specific surface areas, improved active site accessibility, and superior dispersion within reaction media, all of which contribute to enhanced catalytic efficiency (Shah *et al.*, 2025; Zhou *et al.*, 2025). An additional advantage of magnetic iron oxide nanoparticles is their facile separation and recyclability via external magnetic fields, enabling catalyst recovery without complex post reaction treatments (Lokhat *et al.*, 2015). In this context, magnetic transition metal oxide nanoparticles, particularly magnetite (Fe_3O_4) and hematite (Fe_2O_3), have been widely recognized as promising catalysts for a broad range of chemical transformations (Lokhat *et al.*, 2015).

Typically, iron-based catalysts often require promotion with alkali metals to achieve optimal activity and selectivity (Chernyak *et al.* 2022). Alkali promoters such as sodium and potassium have been reported to play a crucial role in modifying the electronic structure and surface basicity of iron catalysts, thereby enhancing their olefin forming capability (Wei *et al.*, 2017; Chernyak *et al.*, 2022). The generation of light olefins is particularly significant, as olefins constitute key intermediates and essential components of gasoline-range fuels. When integrated with acidic zeolites such as HZSM-5, iron-based catalysts can function synergistically as multifunctional catalytic system. In such systems, olefins produced over alkali promoted iron oxide sites can migrate to the Brønsted acid sites of HZSM-5, where they undergo secondary acid catalyzed reactions including isomerization, oligomerization, cyclization, and aromatization. This tandem catalytic mechanism enables effective molecular upgrading and tailoring of the hydrocarbon product distribution. Consequently, the rational combination of alkali promoted Fe_3O_4 nanoparticles with HZSM-5 zeolite offers a powerful strategy for directing product selectivity toward high-value hydrocarbons, including paraffins, aromatics, naphthenes, olefins, and gasoline-range (C_4 – C_{12}) fractions. Such multifunctional catalytic systems are particularly attractive for waste to fuel and hydrocarbon upgrading processes, as they integrate metal catalyzed cracking and olefin formation with zeolite-driven shape-selective upgrading in a single catalytic framework.

Accordingly, the objective of this study is to systematically investigate the influence of alkali and alkaline earth metal promoters namely: Na, K, and Ca, on the catalytic selectivity of iron oxide nanoparticles supported on HZSM-5 zeolite (Na/K/Ca– Fe_3O_4 /HZSM-5) during the thermo-catalytic cracking of used engine oil into gasoline-range hydrocarbons. The study aims to elucidate how different promoter species modify catalyst performance, product distribution and fuel properties, particularly toward the gasoline. Previous studies have demonstrated the feasibility of converting used engine oil into value added fuels and chemicals using a variety of catalytic systems and processing routes. Nevertheless, most reported investigations have focused on thermal cracking, non-zeolitic catalysts, or conventional acidic materials, with limited emphasis on promoter induced selectivity control. To date, there has been no comprehensive investigation addressing the direct conversion of used engine oil into gasoline-range hydrocarbons using Na/K/Ca– Fe_3O_4 /HZSM-5 multifunctional catalysts.

2.0 MATERIAL AND METHODS

Detailed descriptions of the materials employed, sample collection and pretreatment procedures, catalyst synthesis and characterization, thermo-catalytic cracking of the UEO, analysis of the molecular composition of the cracked liquid product, and fuel properties determination have been reported in our

previous publications (Sahabi *et al.*, 2024a; Sahabi *et al.*, 2024b).

In this study, a Taguchi experimental design implemented in Minitab 19 statistical software was employed to investigate the effects of three key process variables on the cracking of UEO. Specifically, the influence of Na/K/Ca- Fe_3O_4 loading on HZSM-5, reaction temperature, and reaction time on catalyst selectivity toward gasoline-range hydrocarbons was systematically evaluated.

3.0 RESULTS AND DISCUSSIONS

3.1 Molecular Composition of Raw UEO and Cracked Liquid Products

The molecular composition of gasoline plays a pivotal role in determining combustion characteristics, as the oxidation kinetics of the fuel-air mixture are strongly governed by the chemical structure, molecular weight distribution, and functional diversity of its constituent hydrocarbons (Sarathy *et al.*, 2017). Variations in paraffinic, olefinic, naphthenic, and aromatic fractions directly influence ignition delay, flame propagation,

knock resistance, and exhaust emissions. Consequently, gasoline is widely regarded as a highly complex multicomponent system comprising hundreds of aliphatic and aromatic hydrocarbons, together with performance enhancing additives, including oxygenated compounds designed to improve combustion efficiency and emissions control (El-Deeb *et al.*, 2022).

Given this compositional complexity, detailed molecular level characterization is essential for assessing fuel quality and performance. In the present study, the molecular composition of the liquid products derived from UEO cracking was systematically analyzed using GC-MS, a well-established technique for resolving individual hydrocarbon species and assigning carbon number distributions in petroleum derived fuels (Wang *et al.*, 2021). The resulting carbon number distributions and PONA classifications of the liquid fractions are presented in Figures 3.1 and 3.2, respectively. These analyses provide critical insight into the extent of cracking, hydrocarbon transformation pathways, and the overall suitability of the produced liquid fuels for gasoline applications.

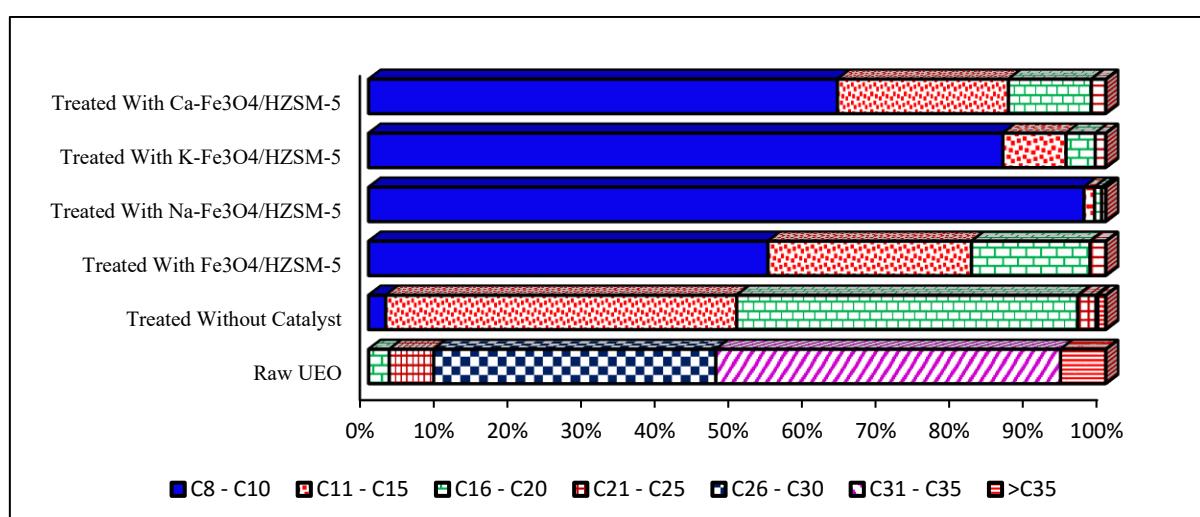


Figure 3.1. Carbon number distribution of raw UEO and the resulting liquid product fractions.

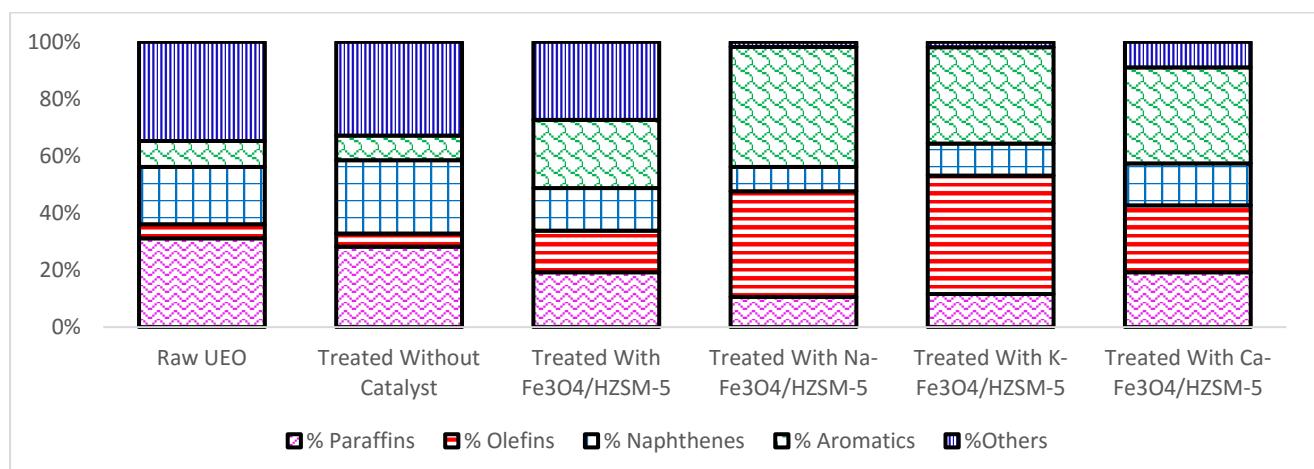


Figure: 3.2: PONA distribution of raw UEO and the obtained liquid product fractions.

As shown in Figures 3.1 and 3.2, the molecular composition of raw UEO is dominated by high-carbon number hydrocarbons in the C₂₆–C₃₅ range, with a predominance of aliphatic species and a minor contribution from aromatic compounds. In addition to hydrocarbons, the raw UEO contains appreciable amounts of oxygenated and halogenated constituents, which are primarily attributed to oxidative degradation of the base oil and additive packages during service life (Singh *et al.*, 2017). After thermal treatment in the absence of a catalyst, the liquid products exhibited a narrower carbon distribution predominately within the C₁₁–C₂₀ range, and the corresponding PONA composition remained similar to that of the untreated UEO, indicating that non-catalytic thermal cracking alone did not affect substantial alteration of hydrocarbon class distribution or achieve substantial upgrading toward gasoline-range fractions.

In contrast, catalytic conversion using iron oxide nanoparticles supported on HZSM-5 zeolite produced a marked shift in product distribution toward lighter hydrocarbons, with a significant fraction concentrated in the C₈–C₁₀ range. The improved selectivity toward lower carbon number hydrocarbons is attributable to the well-defined pore architecture and shape selective properties of the HZSM-5 framework. These structural features facilitate diffusion of bulky UEO molecules into the zeolite channels, where they interact with both Lewis and Brønsted acid sites, enhancing C–C bond scission and formation of lighter hydrocarbon fragments (Liang *et al.*, 2022).

The catalytic treatment also induced a pronounced shift in the PONA distribution toward increased olefinic and aromatic content, indicating that secondary reactions such as cracking, oligomerization, cyclization, and aromatization occurred during the conversion process (Mishra *et al.*, 2021). The emergence of lighter C₈–C₁₀ hydrocarbon fractions, coupled with a reduction in heteroatom containing impurities, suggests the generation of fuel fractions with physicochemical

properties approaching those of commercial gasoline. The synergistic action of Lewis and Brønsted acid sites, in conjunction with the favorable pore structure of the Fe₃O₄/HZSM-5 catalyst, enhances catalytic performance by selectively converting UEO into gasoline-range hydrocarbons with reduced oxygen content via pathways involving hydrogen transfer, catalytic cracking, and deoxygenation (Gong *et al.*, 2023). Overall, the Fe₃O₄/HZSM-5 catalyst demonstrated high efficacy in upgrading long chain hydrocarbons in UEO into lighter, more valuable fuel components.

3.2 FTIR Analysis of Raw Used Engine Oil and Cracked Liquid Products

Fourier transform infrared (FTIR) spectroscopy is a well-established analytical technique widely used for the identification of characteristic functional groups in fuels and hydrocarbon-based materials. The technique is based on the absorption of infrared radiation by molecular bonds, which undergo vibrational transitions at characteristic frequencies that are largely independent of the overall molecular structure (Thanikachalam and Karthikeyan, 2020). As a result, FTIR provides rapid and reliable qualitative information on the chemical functionalities present in complex fuel matrices, including paraffinic, olefinic, aromatic, and oxygen containing species.

Beyond functional group identification, FTIR spectroscopy is also effective for detecting contaminants and residual impurities, such as oxygenated compounds, nitrogen containing species, and additive derived functionalities, which may persist following fuel processing or upgrading (Pinheiro *et al.*, 2017). Consequently, FTIR analysis serves as a valuable complementary technique for assessing chemical changes induced during thermal and catalytic treatment of used engine oil. In the present study, the characteristic absorption bands observed in the FTIR spectra and their corresponding functional group assignments are summarized in Table 3.1, providing insight into the compositional evolution of the fuel samples.

Table 3.1: Absorption Bands of Functional Groups

Band	*V (cm ⁻¹)	Raw UEO	Treated with Fe ₃ O ₄ /HZSM-5	Treated with Na–Fe ₃ O ₄ /HZSM-5	Treated with K–Fe ₃ O ₄ /HZSM-5	Treated with Ca–Fe ₃ O ₄ /HZSM-5
C–H stretch in Alkanes	2990 – 2850	2922.2 2851.4	2953.9 2922.2 2851.4	2965.1 2926.0 2871.9	2961.4 2933.4 2851.4	2953.9 2926.0 2858.9
C=C stretch in Alkenes	2250 – 2100	2100–2200	2100–2200	2100–2200	2100–2200	2100–2200
C=C stretch in Aromatics	1625 – 1440	1455.5	1455.5	1455.5	1455.5	1455.5
–NO ₂ stretch	1390 – 1300	1373.5	1373.5	1377.3	1369.8	1377.3
=C–H stretch In Aromatics & Olefins (out plane)	840 – 600	715.6	805.1	767.8 661.6	814.4 786.5 697.0	814.4 786.5 697.0

The FT-IR spectra of the raw UEO and the cracked liquid products were recorded over the wavenumber range of 4000–650 cm^{-1} to elucidate changes in functional group composition induced by thermal and catalytic cracking. As summarized in Table 3.1, the spectra of the raw UEO and the cracked products exhibit broadly similar absorption features, indicating that the cracking process primarily alters molecular weight distribution rather than completely eliminating the fundamental hydrocarbon functionalities present in the feedstock. The characteristic C–H stretching vibrations of aliphatic hydrocarbons were observed in the range of 2990–2850 cm^{-1} across all samples. The strong absorption intensities in this region confirm that paraffinic hydrocarbons constitute a major fraction of both the raw UEO and the cracked liquid products, consistent with the paraffin rich nature of lubricant base oils (Speight, 2014). Prominent absorption bands detected in the ranges of 2250–2100 cm^{-1} and 1625–1440 cm^{-1} are attributed to C=C stretching vibrations associated with olefinic and aromatic hydrocarbons, respectively. The presence of these bands confirms the generation and retention of olefins and aromatics, which are essential constituents of gasoline-range fuels and play a key role in enhancing octane number (Silverstein *et al.*, 2014).

Further confirmation of olefinic and aromatic structures is provided by medium intensity absorption bands observed in the region of 840–600 cm^{-1} , corresponding to out of plane =C–H bending vibrations. Notably, the liquid product obtained using the Ca– Fe_3O_4 /HZSM-5 catalyst exhibited comparatively stronger absorption intensities in this region, suggesting a higher relative concentration of aromatic and olefinic species. This observation is consistent with the role of acidic zeolite catalysts in promoting secondary reactions such as cyclization and aromatization during cracking (Corma, 1995).

Additionally, a weak absorption band detected at 1748.1 cm^{-1} in the spectrum of the Ca-promoted catalyst product is assigned to carbonyl (C=O) stretching

vibrations, indicating the presence of trace oxygenated compounds. These oxygenated species may originate from partial oxidation of hydrocarbons or from degradation products of lubricant additives that were not completely removed during the cracking process (Pinheiro *et al.*, 2017). The FT-IR spectra also reveal absorption bands in the range of 1390–1300 cm^{-1} in all samples, corresponding to $-\text{NO}_2$ functional groups. These bands are likely associated with nitrogen containing additives, such as nitromb-based detergent or anti wear compounds, originally present in the lubricant formulation.

3.3 Influence of Catalyst Promoters on Gasoline Carbon Range and PONA Selectivity

The influence of different catalyst promoters on product distribution, expressed in terms of paraffins, olefins, naphthenes, and aromatics (PONA), together with the corresponding gasoline-range hydrocarbon fractions, is illustrated in Figure 3.3. The figure depicts the relative contributions of individual hydrocarbon classes within the liquid products and their respective selectivity toward gasoline-range hydrocarbons (C_4 – C_{12}). Such classification is widely employed to evaluate fuel quality and catalytic performance, as PONA composition directly governs combustion behavior, octane rating, and emissions characteristics of gasoline fuels (Speight, 2014; Sarathy *et al.*, 2017). In the present analysis, the identified components were systematically grouped into paraffins (P), olefins (O), naphthenes (N), and aromatics (A), along with an additional fraction comprising heteroatom containing compounds, including oxygen, nitrogen, and halogen bearing organic species. The latter fraction is primarily associated with residual lubricant additives and oxidation by-products present in used engine oil feedstocks and is commonly observed in cracked oil products (Pinheiro *et al.*, 2017). Evaluation of PONA distribution therefore provides valuable insight into the role of catalyst promoters in directing cracking, hydrogen transfer, cyclization, and aromatization reactions toward the selective formation of gasoline-range hydrocarbons.

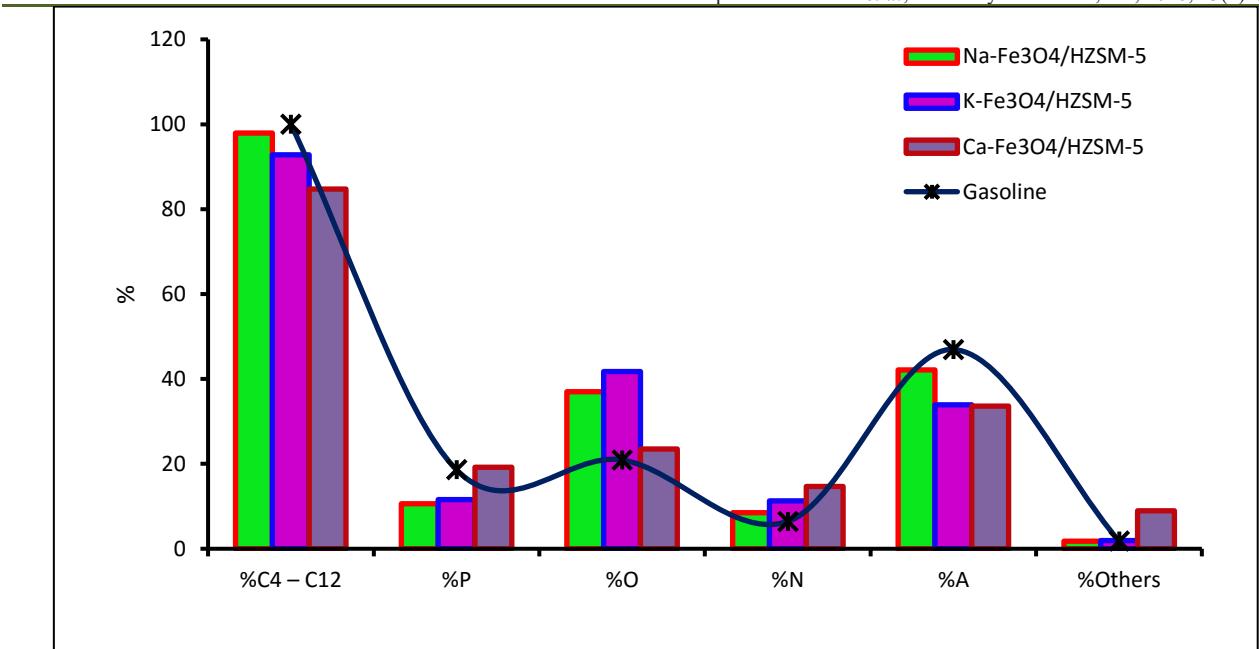


Figure 3.3: Influence of different catalyst promoters on gasoline-range hydrocarbon distribution and PONA selectivity during the catalytic cracking of used engine oil

The results presented in Figure 3.3 clearly demonstrate that the nature of the catalyst promoter plays a decisive role in governing product selectivity during the catalytic cracking of used engine oil (UEO). Across all catalytic systems investigated, the predominant fraction of hydrocarbons produced falls within the gasoline carbon number range (C₄–C₁₂). Compared with the original carbon distribution of UEO, which is mainly concentrated in the heavier C₂₆–C₃₅ range, the product distributions obtained over all promoted catalysts became significantly narrower, with their maxima shifted toward lower carbon numbers. This pronounced shift provides clear evidence of effective cracking of high molecular weight UEO components into lighter hydrocarbons.

Although all four catalysts promoted the conversion of heavy UEO fractions into gasoline-range products, the sodium promoted catalyst exhibited superior performance. In particular, Na-Fe₃O₄/HZSM-5 achieved the highest conversion of UEO to gasoline-range hydrocarbons, reaching 97.91%, a value that closely approaches the hydrocarbon composition of commercially available gasoline fuels. The enhanced selectivity toward gasoline-range hydrocarbons can be attributed to the favorable pore architecture and high density of active acid sites inherent to the HZSM-5 zeolite framework. These characteristics facilitate the diffusion of bulky UEO molecules into the internal pore network, where they interact with both Lewis and Brønsted acid sites, thereby promoting C–C bond scission and the formation of lower molecular weight hydrocarbons (Corma, 1995).

The superior performance of the sodium promoted catalyst is further attributed to the role of

sodium in modifying surface acidity and enhancing catalyst stability. Alkali metal promotion has been shown to regulate acid strength and distribution, suppress excessive secondary cracking, and promote olefin formation, ultimately improving selectivity toward gasoline-range hydrocarbons (Liang *et al.*, 2022; Fatih *et al.*, 2023). These combined effects explain the observed enhancement in gasoline selectivity over Na-Fe₃O₄/HZSM-5 relative to other promoted catalysts.

3.4 Fuel Properties of the Synthesized Liquid Products

Fuel properties constitute critical performance indicators for internal combustion engine applications, as they govern both the compatibility of a fuel with engine components and its combustion behavior within the combustion chamber. Parameters such as ignition quality, energy density, volatility, and distillation characteristics directly influence engine efficiency, power output, fuel economy, and exhaust emissions. Consequently, comprehensive evaluation of these properties is essential for determining the suitability of alternative or upgraded fuels for practical engine operation (Suiuay *et al.*, 2023). In the present study, key fuel properties of the liquid products were determined using standardized analytical procedures in accordance with the specifications of the American Society for Testing and Materials (ASTM). The application of ASTM standard test methods ensures the accuracy, reproducibility, and comparability of the measured fuel properties with those of commercially available gasoline, thereby providing a reliable basis for assessing the potential of the produced fuels for real world engine applications.

3.4.1 Specific Gravity

Specific gravity, commonly referred to as relative density, is a dimensionless parameter defined as the ratio of a fuel's density to that of a reference substance, typically water at a specified temperature. In modern internal combustion engines, fuel delivery systems operate predominantly on a volumetric metering principle rather than direct mass-based control. As a result, fuels with higher specific gravity introduce a greater mass of fuel per injection event under identical

operating conditions. This increased mass flow can contribute to higher engine power output due to the greater availability of combustible material. However, if the fuel's physicochemical properties are not appropriately matched with engine calibration, elevated specific gravity may also lead to increased fuel consumption and higher exhaust emissions, particularly carbon monoxide (CO) and unburned hydrocarbons (UHC), owing to incomplete combustion and locally rich air-fuel mixtures (Deng *et al.* 2018).

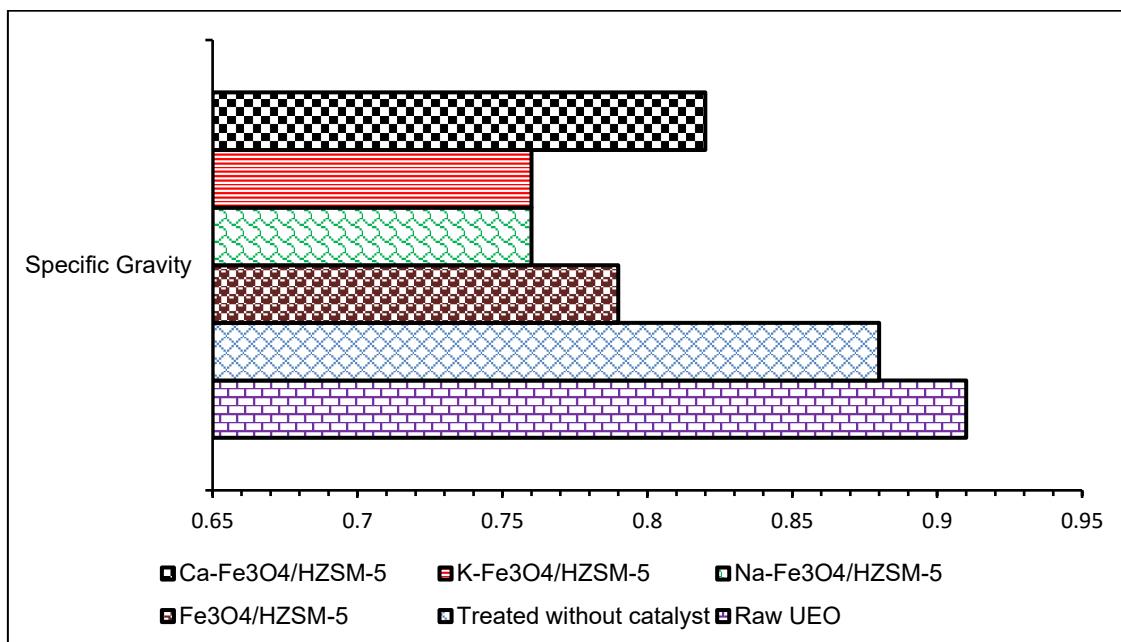


Figure 3.4: Comparison of Specific Gravity of Raw UEO and Cracked Liquid Products

The specific gravities of the raw and treated UEO samples at 450 °C are presented in Figure 3.4. As observed, the specific gravities of all fuel products obtained using the four catalysts fall within the ASTM specified range of 0.75–0.85 for gasoline fuels, indicating their suitability for spark ignition engine applications. Notably, the fuels produced over Na-Fe₃O₄/HZSM-5 and K-Fe₃O₄/HZSM-5 catalysts exhibited specific gravities close to 0.75, consistent with the reported values for premium motor spirit (Ivwurie *et al.*, 2023). Similarly, the fuel derived using Ca-Fe₃O₄/HZSM-5 exhibited a specific gravity of 0.82, aligning with values reported for automotive gas oil (Ivwurie *et al.*, 2023). These results suggest that the choice of catalyst promoter influences the density characteristics of the resulting fuel, with Na and K promotion favoring lighter gasoline-like fractions, while Ca promotion produces slightly denser fractions akin to diesel-range hydrocarbons.

3.4.2 Kinematic Viscosity

Kinematic viscosity is a fundamental fuel property that significantly influences fuel handling, flow behavior, pumping efficiency, and combustion performance. Being a temperature dependent parameter,

viscosity quantifies a fuel's resistance to flow under the influence of gravity and is particularly critical during carburation and injection in internal combustion engines, where it governs spray formation, atomization, and subsequent vaporization within the combustion chamber. Fuels with excessively low viscosity may fail to maintain an adequate lubricating film between moving and stationary components in fuel pumps or carburetors, increasing wear and potentially compromising system longevity. Moreover, low viscosity fuels often exhibit reduced energy density, which can negatively impact overall fuel quality. Conversely, highly viscous fuels tend to atomize poorly, generating larger droplets that hinder complete vaporization and combustion, thereby decreasing thermal efficiency (Kheiralla *et al.*, 2011; Thahir *et al.*, 2019). In addition, incomplete combustion of high viscosity fuels may result in the deposition of coke and other residues within the fuel delivery system, further affecting engine reliability. For this study, the kinematic viscosities of the fuel samples were measured at 40 °C following the ASTM D445 standard, ensuring reproducibility and comparability with conventional petroleum derived fuels.

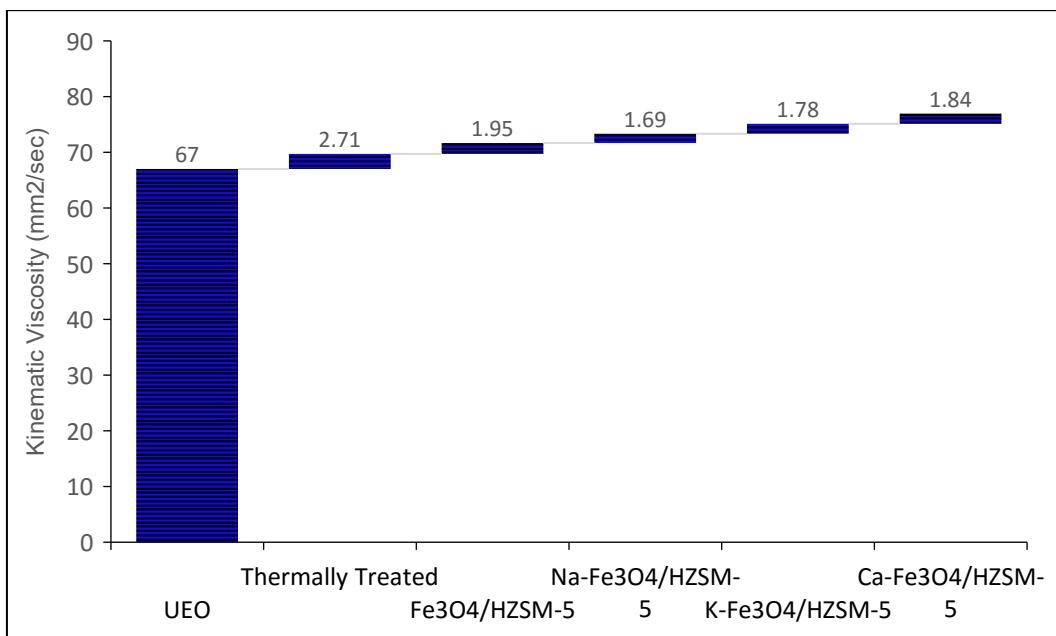


Figure 3.5: Comparison of kinematic viscosities of UEO and cracked liquid products measured at 40 °C

The kinematic viscosities of raw UEO and the cracked liquid products obtained using different catalyst promoters at 450 °C are presented in Figure 3.5. As observed, raw UEO exhibits the highest viscosity of 67 mm²/s, reflecting the presence of long chain, high molecular weight hydrocarbons that hinder flow and increase resistance to deformation (Speight, 2014). The catalytic cracking process significantly reduced the viscosity of all products, with Na-Fe₃O₄/HZSM-5 yielding the lowest kinematic viscosity of 1.69 mm²/s. This reduction is attributed to the efficient cleavage of C-C bonds in heavy hydrocarbons, resulting in lighter gasoline-range fractions (C₄-C₁₂) with lower resistance to flow (Corma, 1995; Liang *et al.*, 2022). The K-Fe₃O₄/HZSM-5 and Ca-Fe₃O₄/HZSM-5 catalysts produced intermediate viscosities, consistent with their respective catalytic efficiencies in cracking and olefin/aromatic formation.

The observed trend indicates that the kinematic viscosity of the fuel products increases with hydrocarbon chain length, consistent with established correlations between molecular weight and flow resistance. All cracked fuel samples exhibited viscosities within the ASTM D445 standard range for gasoline (1.3–2.4 mm²/s at 40 °C), demonstrating their suitability for use in spark ignition engines. Maintaining fuel viscosity within this range ensures proper atomization during injection, efficient vaporization in the combustion chamber, and minimal risk of deposition or coking in fuel delivery systems (Kheiralla *et al.*, 2011; Thahir *et al.*, 2019). Overall, the Na promoted catalyst not only enhanced

conversion of UEO to lighter hydrocarbons but also produced fuel with kinematic viscosity closely matching that of commercial gasoline, highlighting its potential as an effective catalyst for the production of high quality, engine ready fuels.

3.4.3 Flash Point and Autoignition Temperature

Flash point and autoignition temperature are fundamental safety related fuel properties that define the flammability and ignition characteristics of liquid hydrocarbons. The flash point represents the lowest temperature at which a fuel generates sufficient vapor to form an ignitable mixture with air in the presence of an external ignition source, such as a flame or spark (Bagheri *et al.*, 2012). In contrast, the autoignition temperature is defined as the minimum temperature at which fuel vapors spontaneously ignite in air without any external ignition source, providing a measure of the fuel's intrinsic thermal stability (Deng *et al.* 2018; Bagheri *et al.*, 2012).

Accurate determination of these parameters is critical for assessing fuel safety during handling, storage, and transportation. Fuels with higher flash points and autoignition temperatures are inherently safer, as they exhibit lower volatility and reduced susceptibility to accidental ignition under typical operating or ambient conditions. Conversely, fuels with low flash points or autoignition temperatures present higher risks of fire or explosion, particularly under elevated temperatures, making such characterization indispensable for fuel specification and regulatory compliance (Bagheri *et al.*, 2012).

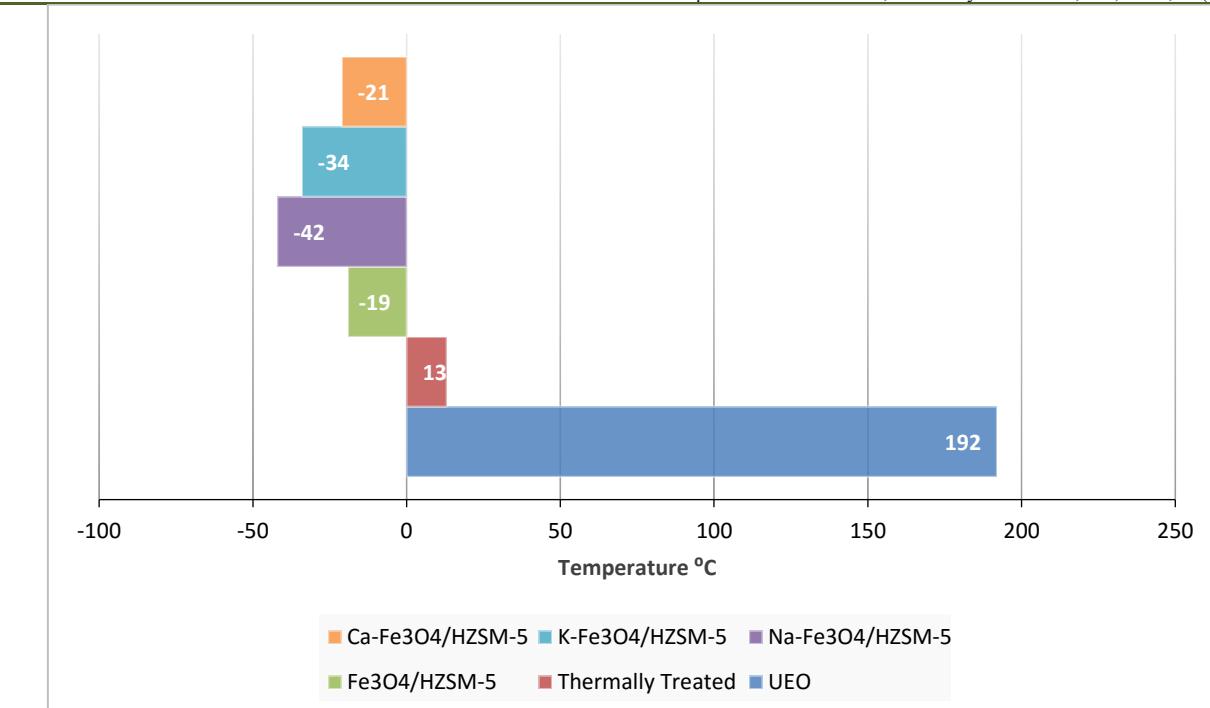


Figure 3.6: Comparison of flash points of UEO and cracked liquid fuel products

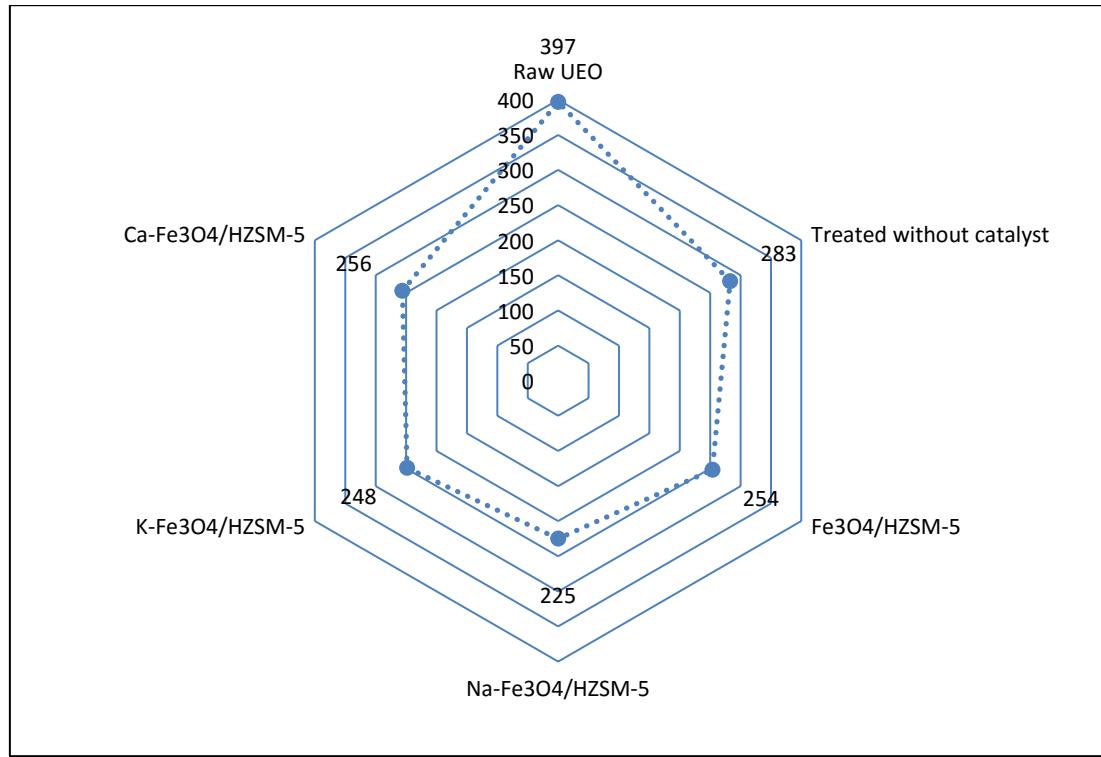


Figure 3.7: Comparison of autoignition temperatures (°C) for raw UEO and cracked liquid products.

Higher flash points and autoignition temperatures serve as key indicators of fuel safety during handling, storage, and transportation, as they reflect lower volatility and a reduced likelihood of unintended ignition under ambient or mildly elevated temperatures (Bagheri *et al.*, 2012). As illustrated in Figure 3.6, the flash points of raw used engine oil (UEO) and the thermally treated product at 450 °C were measured as

192 °C and 13 °C, respectively. The substantially elevated flash point of raw UEO can be attributed to its high content of long chain, heavy hydrocarbons and elevated viscosity, which require greater thermal energy to vaporize combustible components (Speight, 2014; Corma, 1995). Consequently, the raw UEO is inherently safer for storage and transportation compared to the lighter, catalytically cracked fuel fractions, which exhibit

significantly lower flash points and a correspondingly higher flammability hazard.

The autoignition temperatures of the cracked fuel products, shown in Figure 3.7, indicate that all samples exhibit a low propensity for spontaneous ignition, even when exposed to surfaces at approximately 200 °C (Bagheri *et al.*, 2012; Deng *et al.* 2018). This observation suggests that, despite the reduced flash points associated with lighter, gasoline-range fractions, the cracked fuels maintain sufficient thermal stability to mitigate immediate ignition risks under typical handling, storage, and operational conditions. The results underscore the importance of balancing hydrocarbon cracking efficiency with safety considerations, particularly when producing fuels for spark ignition engines.

3.4.4 Octane Number

The octane number (ON) is a fundamental parameter for assessing gasoline fuel quality, as it quantifies the fuel's resistance to engine knock during

combustion. Engine knock refers to the spontaneous and uncontrolled autoignition of the air-fuel mixture in spark ignition engines, which can generate abnormal pressure fluctuations, mechanical noise, and, in severe cases, structural damage to engine components (Sakai & Rothamer, 2023). Knock imposes a practical limitation on engine performance by constraining the maximum permissible compression ratio and preventing operation at optimal combustion phasing under high load conditions. Fuels with elevated octane numbers exhibit greater resistance to detonation, enabling smoother and more efficient engine operation, improved thermal efficiency, and reduced likelihood of engine damage (Sakai & Rothamer, 2023). Accordingly, octane rating is a critical factor in the design and calibration of internal combustion engines, particularly in selecting appropriate compression ratios and ensuring optimal performance across varying operating conditions (Suiuay *et al.*, 2023). High octane fuels are increasingly necessary for modern engines, which often employ high compression ratios to meet efficiency and emission standards.

Table 3.2. Comparison of octane numbers for raw UEO and catalytically cracked fuel products

Sample	Octane Number
UEO	ND
Thermally Treated	86
Fe ₃ O ₄ /HZSM-5	88
Na-Fe ₃ O ₄ /HZSM-5	97
K-Fe ₃ O ₄ /HZSM-5	93
Ca-Fe ₃ O ₄ /HZSM-5	89

High-octane gasoline fuels have been shown to enhance engine performance, improve exergetic efficiency, and reduce emissions of carbon monoxide (CO) and nitrogen oxides (NO_x), particularly in modern spark ignition engines (Khoa & Lim, 2022). Conversely, the use of low octane fuels can be detrimental, especially in engines designed with high compression ratios, where insufficient resistance to autoignition can lead to knocking, reduced efficiency, and potential engine damage.

The research octane numbers (RON) of the liquid products obtained via catalytic cracking of UEO at 450 °C are summarized in Table 4.5. Among the catalysts investigated, Na-Fe₃O₄/HZSM-5 and K-Fe₃O₄/HZSM-5 yielded products with the highest RON values, reflecting enhanced resistance to engine knock and superior gasoline-like quality. Remarkably, these RON values are comparable to those of commercial fluidized catalytic cracking gasoline (FCCG), which typically exhibits a RON of approximately 98 (Olugbenga, 2023). These results highlight the efficacy of sodium and potassium promoted Fe₃O₄/HZSM-5

catalysts in producing high quality gasoline-range fuels from heavy used engine oil.

3.4.5 Carbon Residue

Carbon residue is a critical parameter that reflects the tendency of a fuel to form coke or solid deposits during combustion. It primarily arises from the thermal decomposition of hydrocarbons and the presence of inorganic ash content in the fuel. Elevated carbon residue can adversely affect engine performance by increasing deposits in combustion chambers, altering fuel viscosity and color, and degrading cold flow properties, which collectively reduce engine efficiency and may accelerate component wear or failure (Bhangar *et al.*, 2023; Speight, 2014). In fuel characterization, carbon residue is typically expressed as the percentage of residual carbon remaining after controlled evaporation or pyrolysis of a known quantity of fuel under standardized conditions. This measure provides an indication of the propensity of the fuel to generate deposits and is widely employed as a quality index for gasoline, diesel, and other hydrocarbon fuels (Speight, 2014). Minimizing carbon residue is therefore essential to ensure optimal engine performance, fuel stability, and longevity.

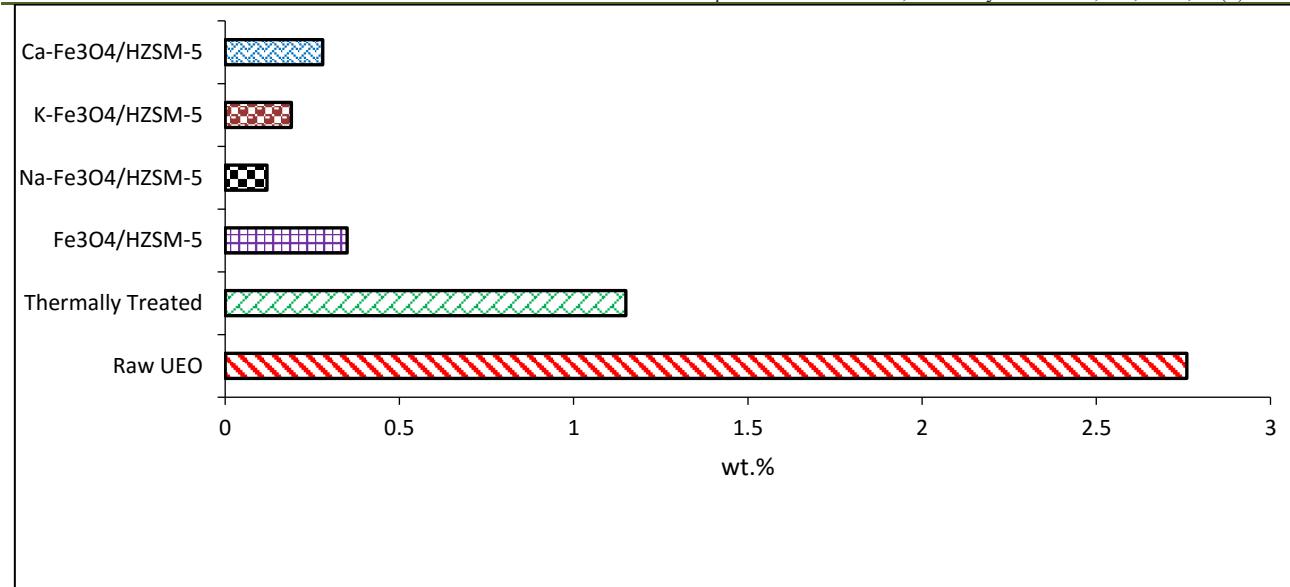


Figure 3.8: Comparison of Carbon Residue of UEO and cracked liquid fuel products.

The carbon residue measurements, illustrated in Figure 3.8, indicate that the untreated UEO exhibits the highest residual carbon content, which can be attributed to the accumulation of unburned hydrocarbons and thermal decomposition products generated during engine operation (Bhangar *et al.*, 2023; Speight, 2014). Elevated carbon residue in the raw UEO reflects its high content of long chain, heavy hydrocarbons and its propensity to form coke deposits, which can adversely affect combustion efficiency and engine longevity.

Following thermo-catalytic cracking, all treated fuel products demonstrated a significant reduction in carbon residue, bringing the values within acceptable limits for gasoline-range fuels. The decrease in carbon residue is primarily associated with the catalytic breakdown of long chain hydrocarbons into lighter, more volatile species, which combust more completely and generate fewer solid residues (Gong *et al.*, 2023). These results highlight the efficacy of Fe₃O₄/HZSM-5 catalysts, particularly when promoted with alkali metals,

in producing cleaner, high quality gasoline-range fuels from heavy UEO feedstocks.

3.5 Influence of Catalyst Loading, Reaction Time, and Temperature on the Selectivity of Cracked Products

An optimization study was carried out using the Taguchi method, as proposed by Genichi Taguchi, on Minitab 19 statistical software (Nagaraja *et al.*, 2023). The objective of this approach was to identify the optimum levels of key process variables namely catalyst loading, reaction temperature, and reaction time, to maximize the performance of the thermo-catalytic cracking of UEO. The influence of these process parameters on gasoline-range hydrocarbon yield and PONA selectivity is summarized in Table 3.3 and illustrated in Figures 3.9 and 3.10. These results provide insight into the relative contribution of each factor and facilitate determination of the optimal conditions for selective production of high value gasoline-range fuels.

Table 3.3. Influence of catalyst loading, reaction time, and reaction temperature on gasoline-range hydrocarbons and PONA selectivity.

Catalyst Loading (wt%)	Temp. (°C)	Time (Min)	Gasoline Range (%C ₄ –C ₁₂)	%P	%O	%N	%A	%Others
5	350	30	87.5119	24.5766	14.7521	22.5065	18.5427	1.8942
5	400	45	93.7109	17.2380	26.8745	15.9523	29.8751	1.7829
5	450	60	97.9143	10.6276	37.0335	8.4953	42.0927	1.7438
10	350	45	88.0523	21.3804	17.3125	20.3077	22.5874	1.8754
10	400	60	93.8540	16.7539	29.3586	14.5687	31.2895	1.7852
10	450	30	97.5421	15.7652	32.9316	13.7259	35.1125	1.7659
20	350	60	88.6187	20.9327	20.3895	19.1078	27.3587	1.8542
20	400	30	93.3084	18.3568	23.5917	16.0568	29.4804	1.7921
20	450	45	97.7365	13.5267	35.7321	11.5117	35.4712	1.7583

P = Paraffins, O = Olefins, N = Naphthenes, A = Aromatics

The catalytic cracking experiments were carried out within a temperature range of 350–450 °C in order to

elucidate the influence of thermal severity on product distribution and selectivity. As evidenced by the results

summarized in Table 3.3, reaction temperature emerged as the most critical parameter governing both the carbon number distribution and the PONA selectivity of the resulting liquid products. The highest conversions of UEO to gasoline-range hydrocarbons were consistently achieved at 450 °C across different catalyst loadings and reaction times, underscoring the dominant role of temperature in promoting effective molecular cracking.

An increase in reaction temperature resulted in a marked enhancement in the selectivity toward gasoline-range hydrocarbons (C_4 – C_{12}), olefins, and aromatics. This trend indicates that key reactions such as C–C bond scission, olefination, cyclization, and aromatization are thermodynamically and kinetically favored at elevated temperatures. Higher temperatures facilitate thermal activation of the catalyst surface, leading to increased accessibility and reactivity of Brønsted and Lewis acid sites, which in turn accelerates hydrocarbon conversion and secondary upgrading reactions (Balboul *et al.*, 2022).

Furthermore, elevated temperatures promote the formation of high energy free radicals, which play a central role in chain scission and cracking mechanisms during the conversion of heavy hydrocarbons into lighter fractions (Mousavi *et al.*, 2022). In contrast, operation at lower temperatures may limit cracking efficiency and favor the accumulation of residual coke on the catalyst surface, resulting in partial blockage of active sites and subsequent catalyst deactivation.

Comparable temperature dependent behavior has been widely reported in the literature. Balboul *et al.* (2022) observed increased gasoline-range selectivity during the catalytic cracking of used sunflower oil over praseodymium-supported alumina at higher temperatures. Similarly, El-Deeb *et al.* (2022) reported a near-linear increase in light hydrocarbon yield with rising temperature during the hydrocracking of hydrotreated tire pyrolytic oil over Ni–W/MCM-41 catalysts derived from blast furnace slag. Collectively, these findings corroborate the pivotal role of reaction

temperature in maximizing cracking efficiency and tailoring product selectivity toward gasoline-range hydrocarbons.

Analysis of variance (ANOVA) was applied to quantitatively evaluate the statistical significance of the operating parameters influencing gasoline-range hydrocarbon selectivity. As summarized in Table 3.4, statistical significance of the model terms was determined based on the magnitude of the F-values and corresponding p-values, with factors considered significant when $p < 0.05$ at a confidence level of $\alpha = 0.05$ (Nagaraja *et al.*, 2023).

The ANOVA results clearly indicate that reaction temperature is the only statistically significant factor affecting gasoline-range selectivity, as evidenced by its low p-value ($p < 0.05$) and comparatively high F-value. In contrast, the remaining linear terms including catalyst loading and reaction time, as well as all two-way interaction terms were found to be statistically insignificant ($p > 0.05$). This outcome confirms that temperature exerts a dominant influence on the cracking efficiency and product distribution, while the effects of catalyst loading and time are comparatively secondary within the investigated experimental domain.

The adequacy and robustness of the regression model are further supported by the high coefficient of determination ($R^2 = 99.56\%$) and adjusted coefficient of determination (adjusted $R^2 = 98.24\%$). These values indicate that the developed model explains nearly all the variability in the response and that the inclusion of model terms is statistically justified without overfitting. The close agreement between R^2 and adjusted R^2 further demonstrates the reliability of the model for predicting gasoline-range hydrocarbon yield and PONA selectivity under the studied operating conditions. Overall, the ANOVA results validate the suitability of the Taguchi-based optimization model for process evaluation and performance prediction.

Table 3.4: ANOVA results for the effects of process variables on gasoline fuel range selectivity.

Source	DF	Adjusted Sum Square	Adjusted Mean Square	F-Value	P-Value
Model	6	141.541	23.5902	75.42	0.013
Linear	3	88.541	29.5137	94.36	0.011
Catalyst Loading (wt%)	1	0.052	0.0517	0.17	0.724
Temperature (°C)	1	85.017	85.0170	271.81	0.004
Time (Min)	1	0.489	0.4887	1.56	0.338
2-Way Interactions	3	0.552	0.1839	0.59	0.679
Catalyst Load. (wt%)*Temp. (°C)	1	0.046	0.0458	0.15	0.739
Catalyst Load. (wt%)*Time (Min)	1	0.456	0.4564	1.46	0.351
Temp. (°C) *Time (Min)	1	0.343	0.3434	1.10	0.405
Error	2	0.626	0.3128		
Total	8	142.167			

DF = Degree of freedom

This indicates that, upon removal of statistically insignificant terms, the refined regression model (Equation 1), comprising four significant parameters, provides an improved predictive capability compared to the original model. This improvement is reflected in the higher adjusted coefficient of determination, which increased from 98.24% to 98.67%, confirming enhanced

model robustness and reduced overfitting. The refined model therefore offers a more reliable representation of the relationship between process variables and gasoline-range hydrocarbon selectivity.

$$\begin{aligned} \text{Gasoline Range (\% C4 - C12)} \\ = 53.32 + 0.0111 \text{ Catalyst Loading (wt\%)} \\ + 0.09670 \text{ Temp. (}^{\circ}\text{C)} + 0.0225 \text{ Time (Min)} \dots \dots \dots 1 \end{aligned}$$

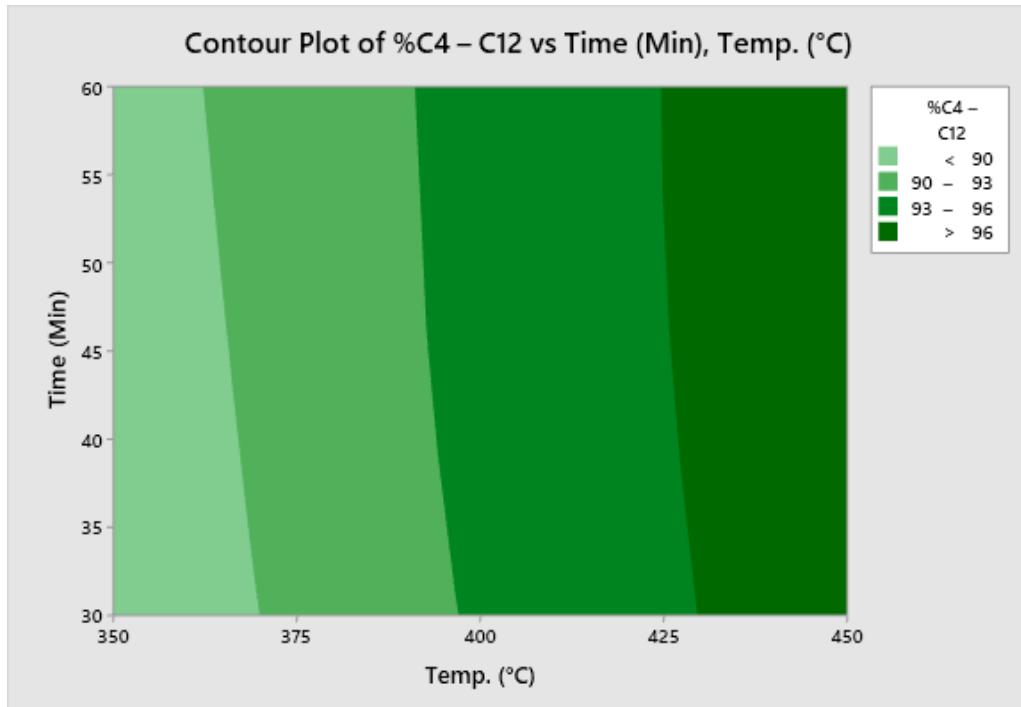


Figure 3.9. Contour plot showing the effect of reaction time and temperature on gasoline-range (C₄–C₁₂) hydrocarbon selectivity

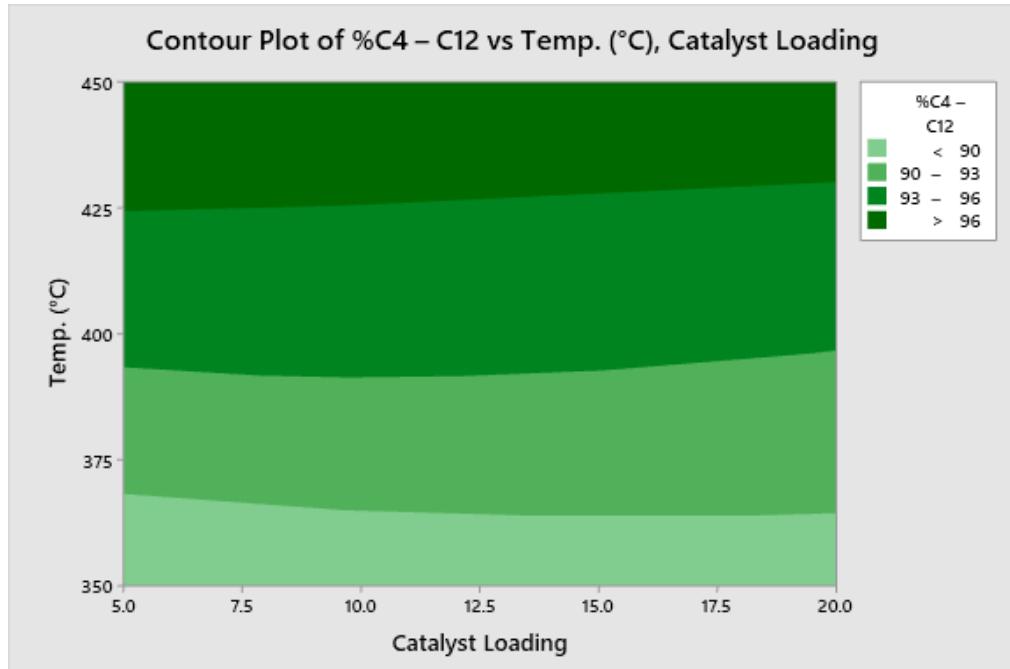


Figure 3.10. Contour plot showing the effect of reaction temperature and catalyst loading on gasoline-range (C_4 – C_{12}) hydrocarbon selectivity

Figure 3.10 depicts the contour plot of C₄–C₁₂ hydrocarbon selectivity as a function of reaction time and temperature. At a fixed catalyst loading, gasoline-range selectivity increased monotonically with increasing temperature, whereas reaction time exerted no statistically significant influence within the investigated range. This observation indicates that, once sufficient residence time is achieved for effective cracking, further prolongation of reaction time does not substantially enhance gasoline-range formation. Similarly, the contour plot illustrating C₄–C₁₂ selectivity as a function of temperature and catalyst loading at constant reaction time further confirms that temperature is the dominant parameter governing gasoline-range hydrocarbon selectivity, while catalyst loading plays a secondary role.

As evident from Figures 3.9 and 3.10, increasing the loading of Na/K/Ca–Fe₃O₄ nanoparticles on HZSM-5 resulted in a gradual decline in selectivity toward gasoline-range hydrocarbons. The maximum C₄–C₁₂ selectivity of 97.91% was achieved using 5 wt.% Na–Fe₃O₄/HZSM-5 (Table 3.3). This superior performance can be attributed to the higher relative proportion of HZSM-5 in the catalyst formulation, which provides a greater density of accessible Brønsted and Lewis acid sites essential for C–C bond scission, isomerization, and secondary cracking reactions at elevated temperatures (Liu *et al.*, 2020).

At higher Fe₃O₄ nanoparticle loadings, catalyst selectivity decreased, which is likely due to nanoparticle agglomeration and partial blockage of the zeolite micropores. Such pore obstruction limits the diffusion of bulky hydrocarbon molecules into the HZSM-5 channel system and reduces the accessibility of active acid sites, leading to underutilization of the catalyst surface and diminished cracking efficiency (Zhang *et al.*, 2019; Wang *et al.*, 2021). These findings highlight the importance of optimizing metal oxide loading to preserve the intrinsic shape selective properties and acidity of HZSM-5, thereby maximizing gasoline-range hydrocarbon production from used engine oil.

4.0 CONCLUSION

This study successfully demonstrated the feasibility of converting UEO into gasoline-range hydrocarbons through thermo-catalytic cracking using iron oxide nanoparticle-modified HZSM-5 catalysts. The integration of Fe₃O₄ nanoparticles with the shape selective HZSM-5 zeolite provided a multifunctional catalytic system capable of effectively upgrading heavy hydrocarbon fractions present in UEO into lighter, high-value fuel components within the C₄–C₁₂ range. Among the catalyst promoters investigated, sodium promoted Fe₃O₄/HZSM-5 exhibited superior catalytic performance, achieving a maximum gasoline-range selectivity of 97.91% under optimized conditions. The enhanced performance of the Na promoted catalyst was attributed to its favourable modification of catalyst

acidity and stability, which promoted efficient C–C bond scission, olefin formation, and subsequent aromatization while minimizing excessive secondary cracking and coke formation. In contrast, higher metal oxide loadings led to partial pore blockage and nanoparticle aggregation, thereby reducing access to active sites and diminishing catalyst selectivity.

Process optimization using the Taguchi experimental design and ANOVA analysis revealed that reaction temperature is the most influential parameter governing product distribution and PONA selectivity, whereas catalyst loading and reaction time exerted comparatively minor effects within the investigated range. The refined regression model, obtained after eliminating statistically insignificant terms, exhibited excellent predictive capability, confirming the robustness and reliability of the optimization approach. Comprehensive characterization of the liquid products confirmed substantial improvements in fuel quality following catalytic upgrading. The cracked fuels exhibited gasoline-like carbon number distributions, favorable PONA compositions enriched in olefins and aromatics, reduced heteroatom content, and improved physicochemical properties, including specific gravity, kinematic viscosity, octane number, and carbon residue. These properties were found to be comparable with those of commercial gasoline, indicating the suitability of the produced fuels for spark ignition engine applications. Overall, the findings of this study highlight the strong potential of Na/K/Ca-promoted Fe₃O₄/HZSM-5 catalysts for sustainable waste to fuel conversion and provide valuable insights into catalyst design and process optimization for upgrading waste lubricating oils. The developed catalytic system offers a promising pathway for mitigating environmental pollution associated with UEO disposal while contributing to the production of alternative transportation fuels.

4. Conflict of Interest

The authors have declared no conflict of interest.

5. Acknowledgements

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